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ORIGINAL ARTICLE

The effects of light curing units and environmental temperatures on C=C conversion of commercial and experimental bonding agents

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Abstract *Background and purpose:* Polymerization of bonding agents (BA) is a critical factor in determining the success of bonded restorations. We aimed to assess the effects of two light curing units and two temperatures on the extent of polymerization (EP) of a commercial BA and an experimental BA.

Methods: Forty BA specimens were randomly divided into 8 subgroups of $n = 5$ to compare the polymerization of two BAs (experimental/Scotchbond) based on the variables: temperature (23/37 °C) and light-curing unit (quartz-tungsten-halogen/light-emitting diode). The EP (%) was measured using differential scanning calorimetry, and analyzed using the *t*-test, two- and three-way analyses of variance (ANOVA), and the Bonferroni test ($\alpha = 0.05$).

Results: There were significant differences between the EP results between the two BAs ($P = 0.012$) and due to the different temperatures ($P = 0.001$), but not between the different light-curing units ($P = 0.548$). The interaction between BA and temperature was significant ($P < 0.001$). The other interactions were nonsignificant.

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Conclusions: The two light-curing units had similar effects on the EP. The EP values were better when curing was performed at human body temperature.

1. Introduction

Bonding agents (BA) are an essential part of composite adhesion (Magne and Douglas, 2000; Peumans et al., 2000; Shafiei et al., 2013). They are used in various clinical applications, such as repairing old composites, veneering laminates on composite restorations, and bonding orthodontic brackets (Jafarzadeh Kashi et al., 2011; Khosravanifard et al., 2010, 2011a). Their use is strongly associated with their ability to prevent microleakage, secondary caries, sensitization, and restoration failure (Shafiei et al., 2013). In addition, their success and longevity are due to clinical conditions, such as the position of the restored teeth, the number of restored teeth in each patient, and the type of the substrate on which the composite is placed (Demarco et al., 2012). The advantageous qualities of BAs are attributed to their degree of conversion (DC) (Antonucci and Toth, 1983; Cotti et al., 2011; Daronch et al., 2005; Jafarzadeh-Kashi et al., 2011; Prasanna et al., 2007), which is the ratio of single carbon–carbon bonds in a polymer matrix to double carbon = carbon bonds in the monomers (Jafarzadeh-Kashi et al., 2011; Prasanna et al., 2007). The clinical importance of BAs justifies the investigation of methods to improve their polymerization. These methods include light curing and warming (Briso et al., 2006; Iriyama et al., 2009; Jung et al., 2001).

Different light-curing systems are used to trigger the polymerization reaction in composite resins, which contain photoinitiators such as camphorquinone (CQ). These systems include conventional quartz-tungsten-halogen (QTH) lamps and solid-state light emitting diodes (LED) (Arrais et al., 2007; Faria-e-Silva et al., 2010). The DC can also be accelerated by warming composite materials (Jafarzadeh-Kashi et al., 2011). Warming the material reduces its viscosity, which enhances radical mobility and increases the collision frequency of unreacted active groups and radicals (Cotti et al., 2011; Daronch et al., 2005; Faria et al., 2010; Jafarzadeh-Kashi et al., 2011; Prasanna et al., 2007).

Although BAs constitute a small proportion of a bonded restoration, they are the weak link in the system. BAs may be the most common cause of restoration failure, which might result in marginal discrepancy due to their shrinkage, thermal expansion, or wear (Magne and Douglas, 2000; Peumans et al., 2000). Therefore, it is valuable to investigate the properties of BAs. Moreover, results pertaining to different brands are not necessarily generalizable to others (Jafarzadeh Kashi et al., 2011; Khosravanifard et al., 2011b) because their formulas differ or are undisclosed. Hence, reporting the results pertaining to a successful BA with an available formula would be of use.

Therefore, we aimed to assess the effects of the light source and environmental temperature on the quality of polymerization of two BAs: one commercial and one experimental. Toward this aim, a precise, sensitive approach is measuring the heat generated during polymerization by differential scanning calorimetry (DSC) (Cotti et al., 2011; Jafarzadeh-Kashi et al., 2011). The objectives of this study were to compare the extent of polymerization (EP) of two dentin bonding agents cured with either an LED or QTH light-curing unit (LCU) at either room or body temperature by using DSC. The null hypotheses were that the EP of the two materials would be similar and that there would be no effect of temperature or the LCU.

2. Materials and methods

This study did not involve any humans or animals, and its protocol ethics were approved by the Research Committee of Tehran University of Medical Sciences, Faculty of Dentistry. In this explorative, experimental, *in vitro* study, an experimental BA (Table 1) and a commercial BA (Scotchbond, 3 M, USA) were cured using two different LCUs (LED or QTH) at two different temperatures (23 °C or 37 °C). In total, 40 BA specimens were randomly divided into the above 8 subgroups (n = 5 each) and compared in terms of their EP under these conditions.

2.1. BA preparation

A BA (Scotchbond, 3 M, USA) was purchased and the contents were analyzed. Based on these approximations, an experimental BA was developed. The experimental BA was prepared by repeatedly examining similar materials with slightly different percentages and testing them until the optimum characteristics were obtained in terms of shear bond strength, microleakage, and polymerization capacity. The materials used to prepare the experimental BA and their weight ratios are listed in Table 1. The commercial BA (Scotchbond, Table 1) was prepared according to the manufacturer's instructions. The experimental BA was prepared as follows. First, CQ was dissolved in the (hydroxyethyl)methacrylate (HEMA) monomer. After the complete dissolution of the powder, bisphenol A-glycidyl methacrylate (bis-GMA) was added to the solution and blended. Finally, dimethyl-para-toluidine (DMPT) was added to the final solution (Erfan et al., 2014).

2.2. DSC

One drop of each BA (the experimental and the Scotchbond Multi-Purpose (MP)) was weighed 3 times with a digital scale (Shimadzu, LIBROR AEU-210, accuracy = 0.0001 g). The specimens were placed on DSC aluminum pans (4.5 × 2.0 mm) and then transferred to the sample holder of the instrument. Two different LCUs, an LED (IEC60601-1 class II, type BF, Coltolux LED, Coltene, USA) and a QTH (IEC601-1 class I, type BF, Coltolux 75, Coltene, USA) were used to light cure the specimens. The light output was evaluated using two radiometers (Demetron 910726 LED Radiometer and Optilux Model 100, Kerr, USA). The LED

Table 1 Materials used for the experimental bonding and their weight percentages.

Type of material	Weight (%)	Manufacturer
Bisphenol A-glycidyl methacrylate (Bis-GMA)	0.62	ABCR (Germany)
Hydroxy ethyl methacrylate (HEMA)	0.37	SIGMA (USA)
DL-camphorquinone (DL-CQ)	0.3	Acros Organics (USA)
Dimethyl-*p*-toluidine (DMPT)	0.7	Acros Organics (USA)

unit was set at over 1000 mW/cm^2, and the QTH unit was set at over 800 mW/cm^2.

The specimens were immediately placed on the aluminum pan of the DSC thermal analyzer (DSC-60, Shimadzu, Kyoto, Japan), and transferred to the sample holder of the instrument to perform the isothermal temperature analysis. For each thermal group for each material, the DSC was adjusted to a definite temperature. Afterward, to start the heat flow measurements, the temperature was immediately altered to the programed temperature in 20 s (Cotti et al., 2011). During each session, after fixing the temperature, the BAs were photopolymerized for 30 s using the two LCUs. The light guide was positioned at a distance of 9 mm from the base of the sample chamber. The heat generated during the polymerization of each material and its peak were recorded and graphically illustrated for each temperature condition. Any difference between the temperature of the sample and the reference was measured and appeared as a peak on the recorder. The empty pan was weighed (Shimadzu) three times, and the average was considered its weight. The normalized heat of the exothermic reaction caused by the conversion of the monomers of each specimen was calculated by dividing the whole differential energy by the specimen weight (Kashi et al., 2009).

The molar heat of polymerization of HEMA and bis-GMA are 50 and 100 kJ/mol, respectively. Based on the ratio of bis-GMA/HEMA for the two studied BAs, the molar heat of polymerization for 100% of polymerization conversion is equal to 89.119 kJ/mol. Therefore, the EP was calculated using the following equation (Kashi et al., 2009): EP (%) = $100 \times \Delta H/89.119$.

All tests were performed on the two different BAs (experimental and Scotchbond MP) under two different light sources (LED and QTH) and at two different constant temperatures (23 °C and 37 °C).

2.3. Statistical analysis

The sample size of this study was determined to be 40 specimens to obtain test powers >0.9 using the averages and standard deviations reported by Tanimoto et al. (2005) and based on the following formula ($\alpha = 0.05$, $\beta = 0.1$):

$$n = \frac{(Z_{1-\alpha/2} + Z_{1-\beta})^2 (S_1^2 + S_2^2)}{(\mu_1 - \mu)^2}$$

Descriptive statistics were calculated. An independent samples *t*-test was used to compare the BAs, the effect of temperature, and the effect of the LCU. A two-way analysis of variance (ANOVA) was used to assess the effect of the BA together with either the effect of the LCU or temperature, and to assess the effect of both the LCU and temperature together. The same test was used to assess interactions among these variables. A three-way ANOVA and a Bonferroni post hoc test were used to evaluate all the variables studied and all of their interactions. The level of significance was set at 0.05.

3. Results

3.1. Pairwise comparison

The independent samples *t*-test showed that the difference in the EP due to temperature was significant ($P = 0.007$). However, there was no difference in the effects of the two LCUs on polymerization ($P = 0.682$). The difference between the two BAs on the EP was non-significant ($P = 0.070$, Fig. 1, Table 2).

3.2. Two-way ANOVA for each pair of variables

3.2.1. BA versus temperature

Significant differences existed between the two BAs ($P = 0.014$), between the two temperatures ($P = 0.001$), and between their interactions ($P < 0.001$).

3.2.2. BA versus LCUs

There was no effect of the LCUs ($P = 0.671$) and a non-significant effect of the BA ($P = 0.069$) on the EP. In addition, no interaction existed ($P = 0.262$), indicating that the effects of the different LCUs were similar for both of the BAs.

3.2.3. LCU versus temperature

There was a significant effect of temperature ($P = 0.008$) and no effect of the LCU ($P = 0.657$) on the EP. In addition, there was no interaction between these two variables ($P = 0.415$).

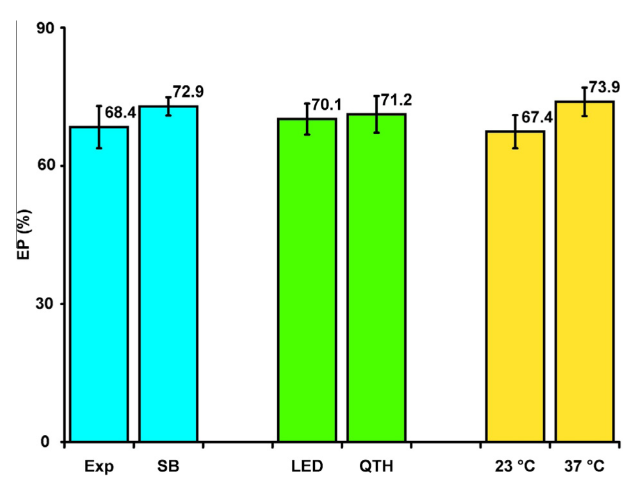

Figure 1 Mean and 95% CI for the EP values (%) in different groups (n of each = 20).

3.3. Three-way ANOVA for all three variables

In the holistic context, the three-way ANOVA showed that there were differences between the results for the two BAs ($F = 7.1$, $P = 0.012$), and the two temperatures ($F = 14.6$, $P = 0.001$). However, there was no difference between the EPs for the two LCUs ($F = 0.4$, $P = 0.548$). The interaction of the variables BA × temperature was significant ($F = 22.6$, $P < 0.001$), indicating that the effect of temperature on the EP of each of the BAs differed from the other one. The interactions between the variables LCU × BA ($F = 2.6$, $P = 0.116$), LCU × temperature ($F = 1.3$, $P = 0.272$), and LCU × BA × temperature ($F = 1.8$, $P = 0.188$) were not significant.

3.3.1. Bonferroni post hoc test

The post hoc test did not detect significant differences between any of the 8 subgroups (Fig. 2).

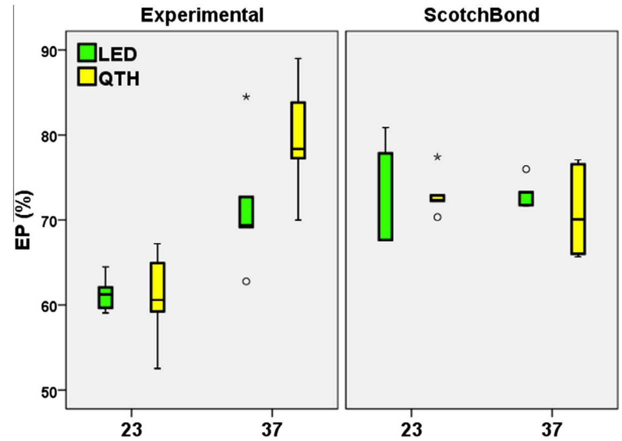

Figure 2 Box plots for the EP values in different subgroups of BA × LCU × temperature (n of each = 5).

4. Discussion

According to the findings of this study, the effects of both the LCUs were similar. Scotchbond showed significantly better results and less variation. Polymerization of the BA at human body temperature resulted in an EP that was approximately 10% better than polymerization at room temperature, and this overall result was associated with the experimental BA. In contrast, Scotchbond showed stable EP values at different temperatures. No specific group contributed to the overall statistical significance; instead, the cumulative differences in all of the subgroups accounted for the significant differences observed because the post hoc test did not detect any significant differences in the pairwise comparisons.

Compared to the successful commercial BA, the experimental BA showed a slightly (but significantly) lower degree of polymerization. Moreover, compared to the commercial material, the experimental BA had greater variability in the results, and its polymerization rate heavily depended on the temperature. These results warrant that more work be done to improve the experimental formula.

The self-limiting cascade of the composite polymerization procedure is constrained by the highly cross-linked polymeric network, which decreases the mobility of reactive monomers (Daronch et al., 2005; Jafarzadeh-Kashi et al., 2011). Therefore, the increased mobility of heated monomers might allow for better polymerization (Jafarzadeh-Kashi et al., 2011). In the present study, although the Scotchbond MP was not significantly affected by temperature, the experimental material was influenced considerably by temperature. Future studies are needed to optimize the experimental formula.

There is debate over the functionality of QTH and LED units. Some authors indicate that the QTH and LED units have a similar, adequate efficacy in polymerizing composites (Uhl et al., 2002). Previous studies indicated that LED efficacy might depend on the light intensity (Briso et al., 2006; Iriyama et al., 2009; Jung et al., 2001). In addition, different QTH units might provide considerably different results (da Silva et al., 2007). In this study, the QTH and LED units were comparable in terms of their effects on the EP. This result was in agreement with that of a previous study (Uhl et al., 2002). However, some investigators reported that BAs cured using LED units might have a lower DC (Arrais et al., 2007). A previous study reported that some LCUs (QTH or LED) might not differ from each other, whereas one LED brand might act more efficiently than another QTH or LED unit (Faria-e-Silva et al., 2010).

The present design was restrained by some limitations. There are limitations over the generalizability of *in vitro* studies to clinical situations. For instance, in clinical conditions, BAs might be light cured for approximately 5–10 s, not 30 s. In addition, a broader range of variables could favor the generalizability of the results. Moreover, the LCU outputs differed in this study. Perhaps future studies should consider this factor, although both light intensities seemed sufficient for a thin layer of transparent BA.

Table 2 Descriptive statistics for the EP values in different subgroups (n of each = 5).

BA	LC	Temp (°C)	Mean	SD	Min	Med	Max	95% CI	
Exp	LED	23	61.31	2.14	59.08	61.25	64.48	58.65	63.97
		37	71.71	8.00	62.78	69.41	84.49	61.78	81.64
	QTH	23	60.90	5.68	52.53	60.60	67.20	53.85	67.95
		37	79.68	7.16	69.99	78.36	88.99	70.79	88.57
SB	LED	23	74.37	6.26	67.65	77.83	80.87	66.61	82.14
		37	73.19	1.74	71.68	73.24	75.98	71.02	75.35
	QTH	23	73.05	2.63	70.34	72.33	77.42	69.78	76.31
		37	71.08	5.51	65.68	70.08	77.07	64.24	77.92

Exp, experimental; SB, Scotchbond; LED, light emitting diode; QTH, quartz-tungsten-halogen; Min, minimum; Med, median; Max, maximum.

5. Conclusion

Within the limitations of this study, it was concluded that the LED LCU had similar effects on the EP of dentin BAs compared with the QTH unit. BAs might be cured more efficiently at human body temperature than at room temperature.

Conflict of interest

The authors declare no conflict of interest.

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References

Antonucci, J.M., Toth, E.E., 1983. Extent of polymerization of dental resins by differential scanning calorimetry. J. Dent. Res. 62 (2), 121–125.

Arrais, C.A., Pontes, F.M., Santos, L.P., Leite, E.R., Giannini, M., 2007. Degree of conversion of adhesive systems light-cured by LED and halogen light. Braz. Dent. J. 18 (1), 54–59.

Briso, A.L.F., Fedel, T.n.M., Pereira, S.r.d.M., Mauro, S.l.J., Sundfeld, R.H., Sundefeld, M.L.c.M.a.M., 2006. Influence of light curing source on microhardness of composite resins of different shades. J. Appl. Oral Sci. 14 (1), 10–15.

Cotti, E., Scungio, P., Dettori, C., Ennas, G., 2011. Comparison of the degree of conversion of resin based endodontic sealers using the DSC technique. Eur. J. Dent. 5 (2), 131–138.

da Silva, G.R., Simamoto-Júnior, P.C., da Mota, A.r.S., Soares, C.J., 2007. Mechanical properties of light-curing composites polymerized with different laboratory photo-curing units. Dent. Mater. J. 26 (2), 217–223.

Daronch, M., Rueggeberg, F.A., De Goes, M.F., 2005. Monomer conversion of pre-heated composite. J. Dent. Res. 84 (7), 663–667.

Demarco, F.F., Correa, M.B., Cenci, M.S., Moraes, R.R., Opdam, N.J., 2012. Longevity of posterior composite restorations: not only a matter of materials. Dent. Mater. 28 (1), 87–101.

Erfan, M., Jafarzadeh-Kashi, T.S., Ghadiri, M., Rakhshan, V., 2014. The effects of dentin bonding agent formulas on their polymerization quality, and together with tooth tissues on their microleakage and shear bond strength: an explorative 3-step experiment. J. Adv. Prosthodont. http://dx.doi.org/10.4047/jap.2014, in press.

Faria-e-Silva, A.L., Lima, A.F., Moraes, R.R., Piva, E., Martins, L.R., 2010. Degree of conversion of etch-and-rinse and self-etch adhesives light-cured using QTH or LED. Oper. Dent. 35 (6), 649–654.

Faria, E.S.A.L., Piva, E., Moraes, R.R., 2010. Time-dependent effect of refrigeration on viscosity and conversion kinetics of dental adhesive resins. Eur. J. Dent. 4 (2), 150–155.

Iriyama, N.T., Tango, R.N., Manetta, I.P., Sinhoreti, M.r.A.C., Sobrinho, L.o.C., Saavedra, G.d.S.F.A., 2009. Effect of light-curing method and indirect veneering materials on the Knoop hardness of a resin cement. Braz. Oral Res. 23 (2), 108–112.

Jafarzadeh-Kashi, T.S., Mirzaii, M., Erfan, M., Fazel, A., Eskandarion, S., Rakhshan, V., 2011. Polymerization behavior and thermal characteristics of two new composites at five temperatures: refrigeration to preheating. J. Adv. Prosthodont. 3 (4), 216–220.

Jafarzadeh Kashi, T.S., Erfan, M., Rakhshan, V., Aghabaigi, N., Tabatabaei, F.S., 2011. An in vitro assessment of the effects of three surface treatments on repair bond strength of aged composites. Oper. Dent. 36 (6), 608–617.

Jung, H., Friedl, K.H., Hiller, K.A., Haller, A., Schmalz, G., 2001. Curing efficiency of different polymerization methods through ceramic restorations. Clin. Oral Investig. 5 (3), 156–161.

Kashi, T.J., Erfan, M., Mirzaii, M., Kalbasi, S., 2009. DSC Evaluation of the extent of polymerization of an experimental bonding agent cured with LED unit. J. Dent. Tehran Univ. Med. Sci. 22 (1), 41–48 (Persian).

Khosravanifard, B., Nemati-Anaraki, S., Faraghat, S., Sajjadi, S.H., Rakhshan, H., Rakhshan, V., 2011a. Efficacy of 4 surface treatments in increasing the shear bond strength of orthodontic brackets bonded to saliva-contaminated direct composites. Orthod. Waves 70 (2), 65–70.

Khosravanifard, B., Nemati-Anaraki, S., Nili, S., Rakhshan, V., 2011b. Assessing the effects of three resin removal methods and bracket sandblasting on shear bond strength of metallic orthodontic brackets and enamel surface. Orthod. Waves 70 (1), 27–38.

Khosravanifard, B., Rakhshan, V., Saadatmand, A., 2010. Effects of blood and saliva contamination on shear bond strength of metal orthodontic brackets and evaluating certain methods for reversing the effect of contamination. Orthod. Waves 69 (4), 156–163.

Magne, P., Douglas, W.H., 2000. Cumulative effects of successive restorative procedures on anterior crown flexure: intact versus veneered incisors. Quintessence Int. 31 (1), 5–18.

Peumans, M., Van Meerbeek, B., Lambrechts, P., Vanherle, G., 2000. Porcelain veneers: a review of the literature. J. Dent. 28 (3), 163–177.

Prasanna, N., Pallavi Reddy, Y., Kavitha, S., Lakshmi Narayanan, L., 2007. Degree of conversion and residual stress of preheated and room-temperature composites. Indian J. Dent. Res. 18 (4), 173–176.

Shafiei, L., Mojiri, P., Ghahraman, Y., Rakhshan, V., 2013. Microleakage of a self-adhesive class v composite on primary and permanent dentitions. J. Contemp. Dent. Pract. 14 (3), 461–467.

Tanimoto, Y., Hayakawa, T., Nemoto, K., 2005. Analysis of photopolymerization behavior of UDMA/TEGDMA resin mixture and its composite by differential scanning calorimetry. J. Biomed. Mater. Res. B Appl. Biomater. 72 (2), 310–315.

Uhl, A., Mills, R.W., Vowles, R.W., Jandt, K.D., 2002. Knoop hardness depth profiles and compressive strength of selected dental composites polymerized with halogen and LED light curing technologies. J. Biomed. Mater. Res. 63 (6), 729–738.